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Characterization of Stickie Contaminants From OCC Recycle Mills

P. Gupta, K.T. Hodgson, A.E. Emery, and T.J. Heindel

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CHARACTERIZATION OF STICKIE CONTAMINANTS FROM OCC RECYCLE MILLS

Pramit Gupta and Kevin T. Hodgson
Paper Science and Engineering
University of Washington
Seattle, WA 98195-2100

Adele E. Emery and Theodore J. Heindel
Institute of Paper Science and Technology
500 10th Street, NW
Atlanta, GA 30318-5794

ABSTRACT

Contaminant removal is essential to convert recovered paper into a reusable fiber. It has a direct bearing on the yield from recovered paper and total costs. Stickie contaminants, a collection of tacky, adhesive-like materials, are considered to be one of the greatest barriers to increasing secondary fiber usage across nearly all paper grades. This research effort aims to identify those stickie contaminants that are currently not being removed by traditional OCC recycle unit operations, and to identify and optimize processes to more successfully remove these troublesome contaminants from the OCC process stream.

An important first step in the identification of stickies is to characterize them on the basis of their size. A selective dye to stain stickie particles was identified and a handsheet bleaching and staining procedure was developed to easily identify hydrophobic particles when performing image analysis of OCC stock. Although preliminary results showed that some particles can be found in the water fraction of stock samples, the majority of the particles found in actual OCC pulp samples remain with the fiber fraction and correspond to sizes (20-500 μm) that are amenable to removal by dispersed air flotation.

INTRODUCTION

By the year 2000, the paper industry will be recovering approximately 50% of all the paper and paperboard consumed in the United States. The growth in recovered paper use has been both to replace virgin wood pulp for economic considerations and for environmental concerns. The growing utilization of secondary fiber across all paper grades introduces a wide variety of contaminants to the input stream. It is estimated that the total cost of these contaminants in lost production time, raw material, final product downgrading, and landfill costs could be as high as $650 million annually within the United States [1].

The type of contaminants classified under the general term of "stickies" are especially important. They refer to either a single component contaminant or an agglomeration of several contaminants that stick to the paper machine, final product, or one another. Stickies can be generally classified as hydrophobic, deformable materials of various solubilities and melting points. Most stickies are relatively insoluble in water over a wide pH range and behave as solid particles in the system. The hydrophobic nature of stickies represents a driving force to escape the water phase and deposit onto other hydrophobic surfaces such as forming fabrics, press felts, and roll covers. A density similar to wet fiber [2] prevents their efficient removal by centrifugal cleaning. The thermoplastic nature of these particles and their tendency to change shape and form with temperature limits their removal by screening.

Stickies comprise a wide variety of contaminant chemistries that include hot melt glues, pressure sensitive adhesives, waxes, thermoplastic resins, inks, UV lacquers, and coating binders. They may exhibit their original chemical and physical characteristics, or be a combination of the aforementioned materials possessing new characteristics. It should be noted that the composition of these materials is complex to begin with, and becomes much more so after residing and agglomerating in a paper machine environment. The most notable complexity is that stickie deposits from a paper machine commonly contain a significant fraction (20-30%) of inorganic materials, such as calcium carbonate or clay [3, 4]. These become entrained in the deposit during the residence time in either the recycling or papermaking process. Besides the main

elastomer polymer, adhesives also contain small amounts of tackifiers such as gum rosin and poly (terpene) resins. These tackifiers are often obtained as byproducts of wood pulping operations.

Despite much research and advances in equipment, stickies remain a problem for a variety of reasons [5]. They can prevent good fiber-to-fiber bonding and increase the risk of web breaks [6]. Stickies reduce product quality due to issues such as picking, pinholes, bleed through, appearance, and soft rolls [7]. They result in premature replacement of wires and felts and reduce converting and printing process efficiency. Stickies can also build up in the approach piping and in closed white water systems.

The intolerance to even low levels of stickie contamination in many grades and the complex chemical nature of stickies make it crucial that a total system strategy be used in their control. Unfortunately, due to the aforementioned wide variety of stickies and their characteristics, a stickies problem solved in one area of the machine can be transferred to another area of the papermaking process. Experience has shown that a combination of treatments is usually needed for effective control of stickie-related problems. Mechanical treatments in a typical old corrugated container (OCC) operation include repulping, stock screening, cleaning, and dispersion [8, 9]. The surface energy of a stickie may be chemically modified from a low surface energy (i.e., hydrophobic) to a high surface energy (i.e., hydrophilic) by adsorbing a polymeric surfactant onto its surface [10]. The induced hydrophilic characteristics reduces the tackiness and agglomeration of stickie particles, which in turn reduces machine deposition and improves product quality. Other control techniques include chemical dispersion, detackification, wire treatment, passivation, and cationic fixation [9].

Image Analysis has been used extensively over the years for evaluating and quantifying contaminants in papermaking. Handsheets are examined by image analysis to obtain information on the size distribution of contaminants. Jean and Nguyen [16] used image analysis to quantify stickies in OCC fibers and suggested modifications to remove shives and lignin from the OCC pulp. The technique required stickies to be on the surface of handsheets and procedures to enhance stickie diffusion to the handsheet surface were utilized. Zeyer et al. [17] evaluated the performance of a scanner for image analysis. The biggest disadvantage of a scanner is its limitation in resolution. Insufficient resolution can lead to either over or underestimation of the size of a spot, depending on the gray scale value that is used for its detection. Compared to an image analysis system using a microscope or a macrolens on a camera, the major advantages of the scanner are comparatively low costs, ease of use, and the possibility to analyze larger paper surface areas. Yordan and Maat [18] used a scanner-based image analyzer to determine the efficiency of talc as a pitch and stickies control agent. In general, image analysis involves some or all of the following steps: calibration, image acquisition, image discrimination, image alteration, elimination of negligible particles, particle filling, particle reconstruction, particle analysis, and interactive tracing [19]. The images acquired using a camera are transmitted to a computer that stores, processes, and measures the various characteristics of these images. Repeatability of image analysis results can potentially be operator-dependent, which may affect the final results quantitatively.

An important point to be considered during image analysis is to be able to distinguish stickie particles from other small particles which can be present in real pulp samples, such as fines, fillers, and detached small ink particles. Fiber fines and other extraneous small particles in real-world pulp samples can be a significant interference in trying to count stickie particles. Contrast between fibers and contaminants can be improved using a water-soluble dye to color the fibers or by using hydrophobic dye to color the contaminants. In either case, what are essentially measured are the concentration and the size distribution of hydrophobic contaminants that may or may not be stickies.

Stickies, thus are one of the greatest obstacles to increasing secondary fiber usage. The overall objective of this joint research effort is to separate and remove stickie contaminants more completely from OCC fiber streams during the recycling process. Currently, the removal techniques of screening and centrifugal cleaning are chiefly employed to remove unwanted materials in paper grades where OCC is the primary fiber source. Although typically 95-99% of stickie contaminants can be removed with screens and cleaners, the small amount that remains can still cause serious problems. Typically, it is much more cost-effective to eliminate stickies early in the process. If a greater fraction of these particles is prevented from reaching the paper machine in the first place, the deposition and product quality problems they currently cause should be either greatly reduced or eliminated altogether. In order to maximize the results of this approach, it is first necessary to determine exactly what *size range* of stickie particles is actually causing deposition problems.

This could be colloidal stickies, which agglomerate and deposit throughout the process; "macrostickies", which either deposit on the machine surface or blemish the finished sheet, or both categories of stickie particles could be of significant importance. Once the size range of these particles has been determined, separation and removal strategies can be considered. However, determining the size range of the problematic particles is an important, but not a sufficient step, in investigating methods for their removal. Other particle properties such as particle shape and density, surface energy, and electrostatic potential (zeta potential) are also keys to proceeding towards an effective removal technology.

For colloidal and near-colloidal size particles (less than about 10 μm in effective diameter), one or more washing stages should be effective in removing these contaminants from a recycled OCC fiber stream. Subsequent water clarification could be addressed by dissolved air flotation (DAF) because of its known effectiveness in clarifying wastewater streams in both OCC and deinking plants. Hydrophobic particles in the size range of approximately 20 μm to as high as 400 μm are much more efficiently removed by dispersed air flotation. Stickies as mentioned previously usually have hydrophobic surface properties. This suggests that dispersed air flotation may be a promising operation to remove these contaminants from brown paper grades if their size range is appropriate [11-15]. Dispersed air flotation is currently used to remove ink particles, which generally also have hydrophobic surface properties.

EXPERIMENTAL PROCEDURES

A sampling procedure was initiated with an OCC recycling mill. Pulp samples were obtained from the high-density storage chest, the last point between the OCC process and the paper mill plant after all the current contaminant removal operations had been utilized. Hence, any contaminants found in these samples will be passed directly on to the paper machine. Cleaned OCC stock was obtained on two different days in January 1997. Three days separated the sampling, which would allow for some furnish variability. Another batch of samples was obtained in August 1997. All samples were kept in a refrigerator to reduce particle agglomeration. Analysis was performed at room temperature.

The samples obtained were separated into fiber and filtrate fractions in order to focus on small particles which would be suspended in the filtrate and not attached to fiber or fines. The separation was accomplished using a Bauer-McNett classifier. A Coulter Counter (a particle-sizing device) was used to obtain the distribution of the particles in a liquid phase with respect to their size and volume.

Image analysis software (Optimas, Bioscan Corp., Edmonds, WA) was used to characterize the contaminants in the fiber fraction sample. Particle size characterization attempted with image analysis of unaltered OCC handsheets proved unsuccessful. The presence of other small, suspended particles, such as shives, made it very difficult to distinguish stickie particles as a stand-alone component in the examined samples. We then investigated the use of potential dyes that would selectively stain stickies and not cellulosic material. This would increase the contrast between the fiber and stickie particles to allow differentiation when performing image analysis. Since there are many different types of stickies, it is not practical to identify stains for each type. However, the majority of stickies are hydrophobic in character. Therefore, in developing this procedure, we focused on those stains that were specific for hydrophobic particles.

OCC samples were artificially spiked with a curtain coating wax (Astor 3040) and a hot melt by "painting" melted wax and hot melt on a linerboard sheet, allowing it to cool, and then disintegrating the sheet at room temperature. Handsheets were made from this stock and were stained with five different stains as possible candidates for staining hydrophobic particles [20]. After these initial trials, it became apparent that fiber bleaching would also be necessary to increase the contrast level between the contaminants and the fiber. Staining trials were performed for various time periods and at two different temperatures (~22°C and ~37°C) to identify the best sample preparation sequence. In general, the best results were obtained when the fiber was bleached and then stained with Sudan IV (a red stain) at elevated temperatures (~37°C) for an extended time period (typically 24 hours). Details of the bleaching and staining procedure used in this study are given in the appendix, and include the following major steps:

1. Bleach the fiber sample at room temperature
2. Make thin handsheets with a basis weight of approximately 20-25 g/m^2

3. Stain the handsheets with Sudan IV
4. Perform image analysis on the handsheets

Although it could be seen visually that there were some black specks (likely to be ink particles) that would be counted along with the red stained particles, these were infrequent enough that they were assumed to have a negligible effect on the final results. The image analysis software records the area of the particle, and the equivalent particle diameter (defined as the diameter of a circle with the same particle area) is determined from this reading. The particle sizing results were then manipulated using an Excel macro written to classify them into "bins" from 50 to 500 μm in increments of 50 μm. A sample field from one of the handsheets, obtained with a magnification of 6X, is shown in Figure 1 as a reference.

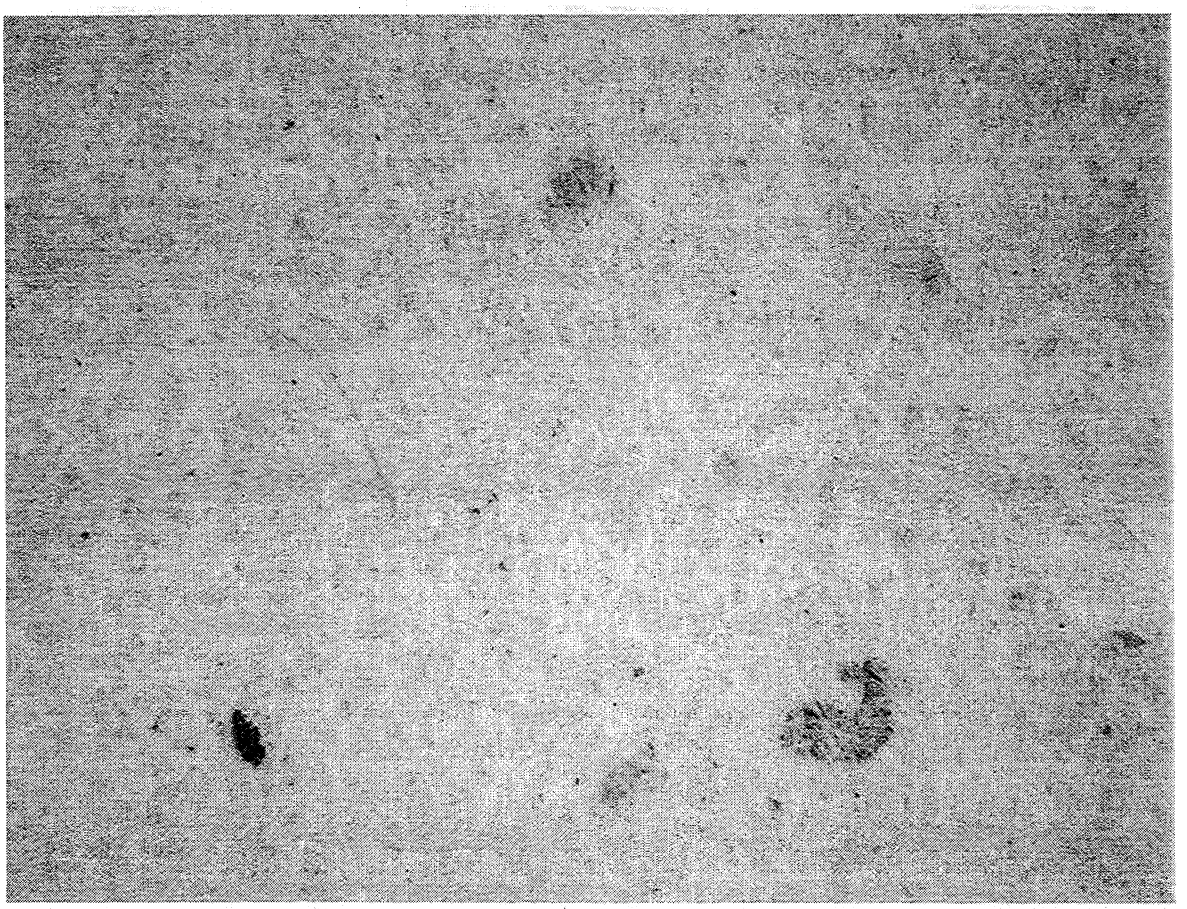

Figure 1: Sample image field (8.4 mm x 6.3 mm) showing contaminants in "clean" OCC.

RESULTS

A. Validation of Bleaching/ Staining Procedure on Particle Size

Stickie particles may agglomerate as pH is raised [21], so experiments were performed to determine if the bleaching procedure used in this work has a significant effect on contaminant size. This was necessary since the hypochlorite bleaching procedure used raised the pH of the stock to approximately 9. To accomplish this, a wax (Astor 3040) of a known particle size distribution was created following the procedures of Bose et al. [22] and then added to a clean unbleached kraft pulp. The bleaching procedure used in this program was then used in this wax/fiber slurry. After staining the handsheets with Sudan IV, no particles were found trapped in the handsheets. The particles found in the *filtrate* from the bleaching and washing step were then analyzed. As shown in Table I, the average particle size found in the filtrate was the same as that of the original particles. Additionally, the particle size distributions before and after bleaching were nearly

identical (Figure 2). Therefore, the bleaching step used to enhance the image analysis contrast does not appear to alter the wax particle size distribution.

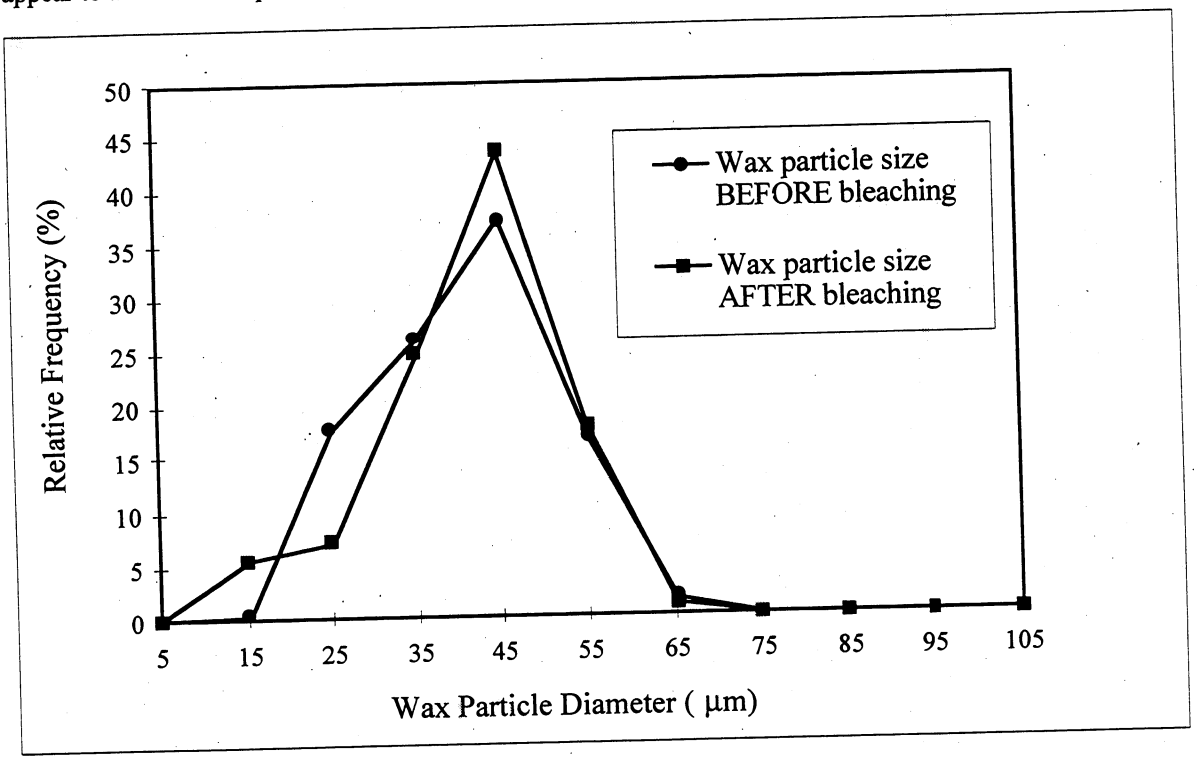

Figure 2: Wax particle size distributions before and after bleaching.

Table I: Wax particle sizes before and after bleaching.

Wax Particle	BEFORE Bleaching	AFTER Bleaching
Median Particle Size (μm)	31.3	32.8
Average Particle Size (μm)	30.7	31.2
Standard Deviation (μm)	10.2	10.6
Variance (μm^2)	100	110

Due to the significant variability of actual stickie particle composition, it is not practical to evaluate the effect this bleaching procedure has on all potential stickie particles found in OCC. However, wax is known to be a common component in OCC stickie particles, both by itself and as a major component of some hot melt adhesives. Therefore, it was felt that wax is a reasonable model to determine the effect bleaching has on stickie particle size. With the original wax particle size distribution used in the bleaching experiment, it was not possible to determine if the staining procedure itself has an effect on the contaminant particle size. However, we believe the effect should be negligible. One reason for this is that the contaminant particles are "locked" in place in the handsheet during the staining procedure. Therefore, particle agglomeration and/or breakup should be negligible. There is a small possibility that a contaminant particle may be soluble in the Sudan IV solution. For this to significantly affect the particle size, it would have to be very soluble in Sudan IV, which is highly unlikely [20].

B. Particle Size Analysis of OCC Mill Samples

A "finished" OCC filtrate sample was analyzed with the Coulter Counter. First, a background particle count in potential suspending liquids (e.g., standard electrolyte, tap water, and distilled water) was taken. The results indicated that these liquids contain particles slightly larger than colloidal in size, with few, if any, particles greater than about 10 μm. In the filtrate sample, about 20% of the particles were spread over the range of 4-30 μm. The remaining 80% of the particles were in the size range of 2-4 μm, which is also

the range of the particles found in the "blank" samples. Since the filtrate sample had many particles in the size range of the "blank" sample, and the Coulter Counter could not distinguish between contaminants, fines, and the background particles in the suspending liquid, it was decided to focus our attention on the fiber fraction. Viewing the filtrate under a light microscope reinforced this decision; most particles in the filtrate appeared to be fines, and few, if any, stickie contaminants were observed.

For the fiber fraction, thin handsheets were bleached and stained, and image analysis was performed as outlined in the procedures section. Preliminary contaminant particle size distributions were obtained from OCC samples taken in January 1997. The results for the samples obtained in the month of August (hereafter referred to as "Aug-C") are summarized in Table II and Figure 3.

Table II: Summary of the particle size found in the August 1997 fiber fraction samples.

Sample	Aug C-1	Aug C-2	Aug C-3	Aug C-4
Number of Handsheets	3	4	3	3
Total Number of Fields Analyzed	12	16	12	12
Total Field Area (cm^2)	33.6	44.8	33.6	33.6
Total Contaminant Area (cm^2)	0.22	0.33	0.16	0.17
Average Particle Diameter (μm)	112	118	110	116

The image analysis involved a fixed region of interest of 2.8 cm^2 for each field, corresponding to a fixed magnification of 5X. At this magnification, the lower limit of particle resolution was approximately 25 μm. Particle size distributions are reported in terms of number frequency of a given size range per total unit area analyzed. The x-axis in Figure 3 is read in the following manner: the bin marked "25" represents particles in the 0-50 μm size range; the bin marked "75" represents particles in the 50-100 μm size range, etc. The last bin marked "525" represents those particles with an equivalent diameter greater than 500 μm. Table II shows that each sample has a similar average particle size. Variations in the total contaminant area can be seen, implying different levels of dirt in each of them. But the samples have the same general particle size distribution, with a peak in the 50-100 μm range (Figure 3).

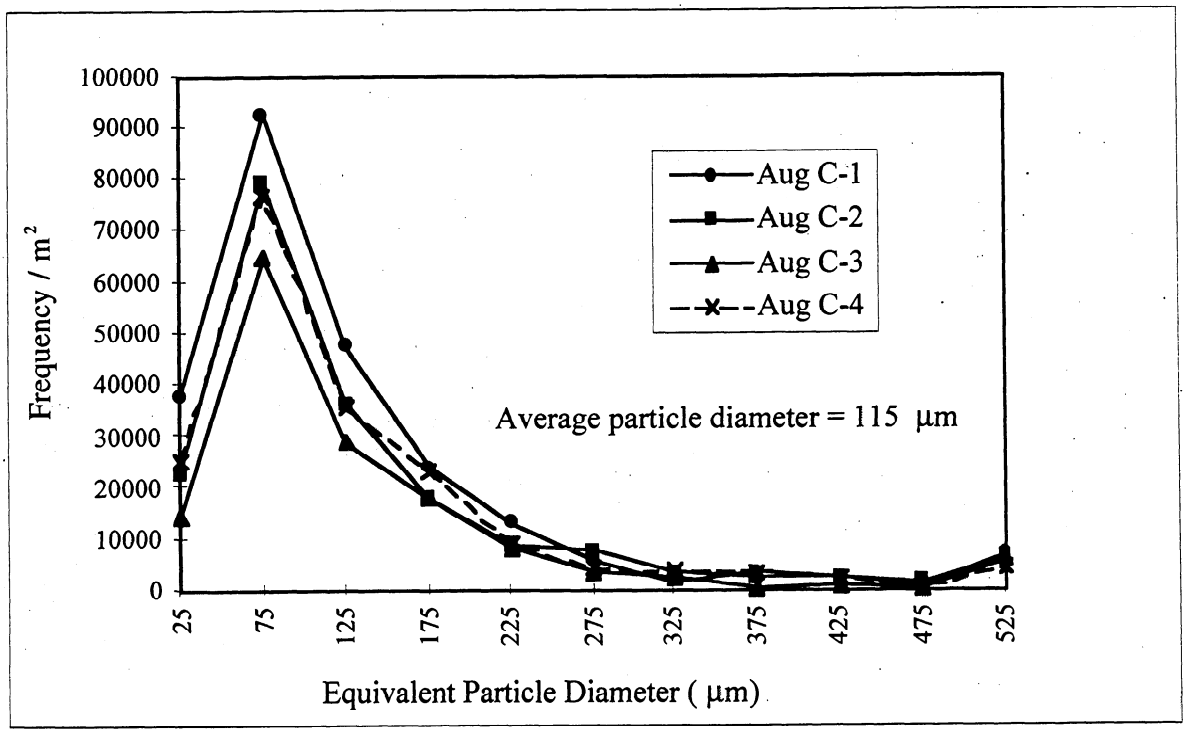

Figure 3: Particle size distribution for "Aug-C" samples.

For both the January and August samples, although there were differences in the total contaminant area, the particle size distribution and the average particle diameter were quite comparable. Figure 4 summarizes the contaminant particle size distribution for all data sets collected; they all show a peak in the particle size distribution in the 50-100 μm range. The screens and cleaners of present OCC systems quite effectively remove particles larger than about 200-250. The smallest screens available today have slots about 150 μm in size. The passage of some material greater than this size is due to the flexibility and morphology (thinness) of many stickie contaminants (i.e., they are forced through the screens). When they become about twice the diameter of the screen opening (~300 μm), the large majority appear to be successfully removed. Thus, we conclude that this analysis is reasonably representative of OCC stickie contamination on an overall basis.

Most importantly, these particle size distributions affirm one of the critical premises put forth for the relevancy of our work. They clearly show that the majority of the particles are well into the range where dispersed air flotation should be the most promising removal technique, and that the particle size is too large for effective removal by washing. If the particle shape and densities were accurately known and the frequency was converted to a mass or volume basis, the distribution peak would shift to an even larger particle diameter (to the right in the previous plots). Flotation has been investigated before for stickies removal, but the emphasis has been on high-grade deinking furnishes [17, 18]. There has not been a concerted effort to our knowledge to specifically tailor flotation for removal of stickies from brown, nondeinked fiber such as OCC or bag stock.

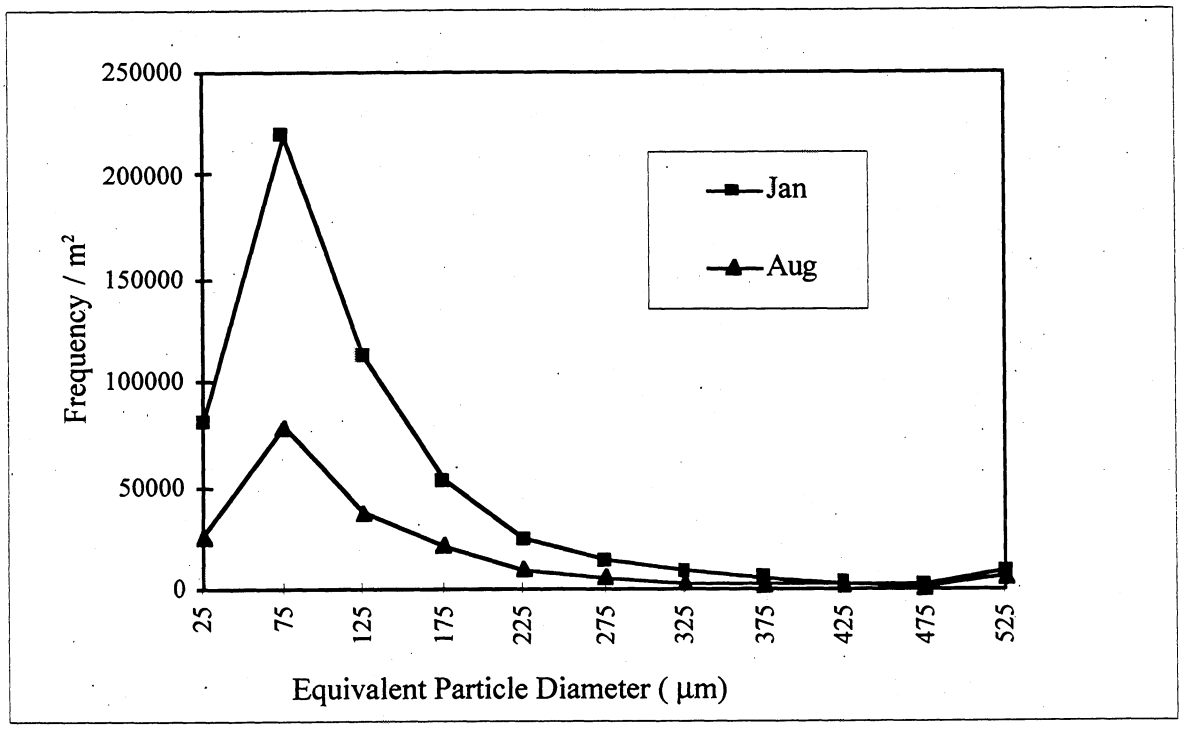

Figure 4: Summary of the contaminant particle size distribution.

Although the particle size analyses reveal a maximum number of particles in the 50-100 μm size range, it should be noted that some very small particles are still present. Viewing the sample handsheets under a microscope at high magnification reveals these small particles, but most are on the order of the fiber diameter or larger (i.e., 20-40 μm). An example of such particles is shown in Figure 5 for a sample handsheet under 125X magnification. The dark regions in this figure (a black and white in this document, but shades of red under the microscope) are the contaminants, while the fiber width is clearly visible and can be used as a rough size indication. As shown, these contaminants are on the order of the fiber diameter, and this size is still well within the acceptable size range for flotation.

CONCLUSIONS

Sample streams from an operating OCC processing plant were collected and analyzed with regard to stickie contaminants present. It was first determined that few, if any, of the contaminants resided solely in the aqueous phase of the samples. This focused the investigation on contaminants present in the fiber fraction. In order to quantify the amount of stickie contaminants present, image analysis of handsheets was selected. Using image analysis to analyze handsheets of brown, unbleached fiber for stickies required the development of a bleaching and staining procedure in order to improve the contrast between particles and fiber.

Samples taken approximately eight months apart were analyzed and number-based particle size distribution generated. These analysis revealed:

1. The general shape of the size distribution was similar for both time periods.
2. The average particle size was nearly the same for both time periods.
3. The vast majority of the particles counted were between about 50 and 200 μm.

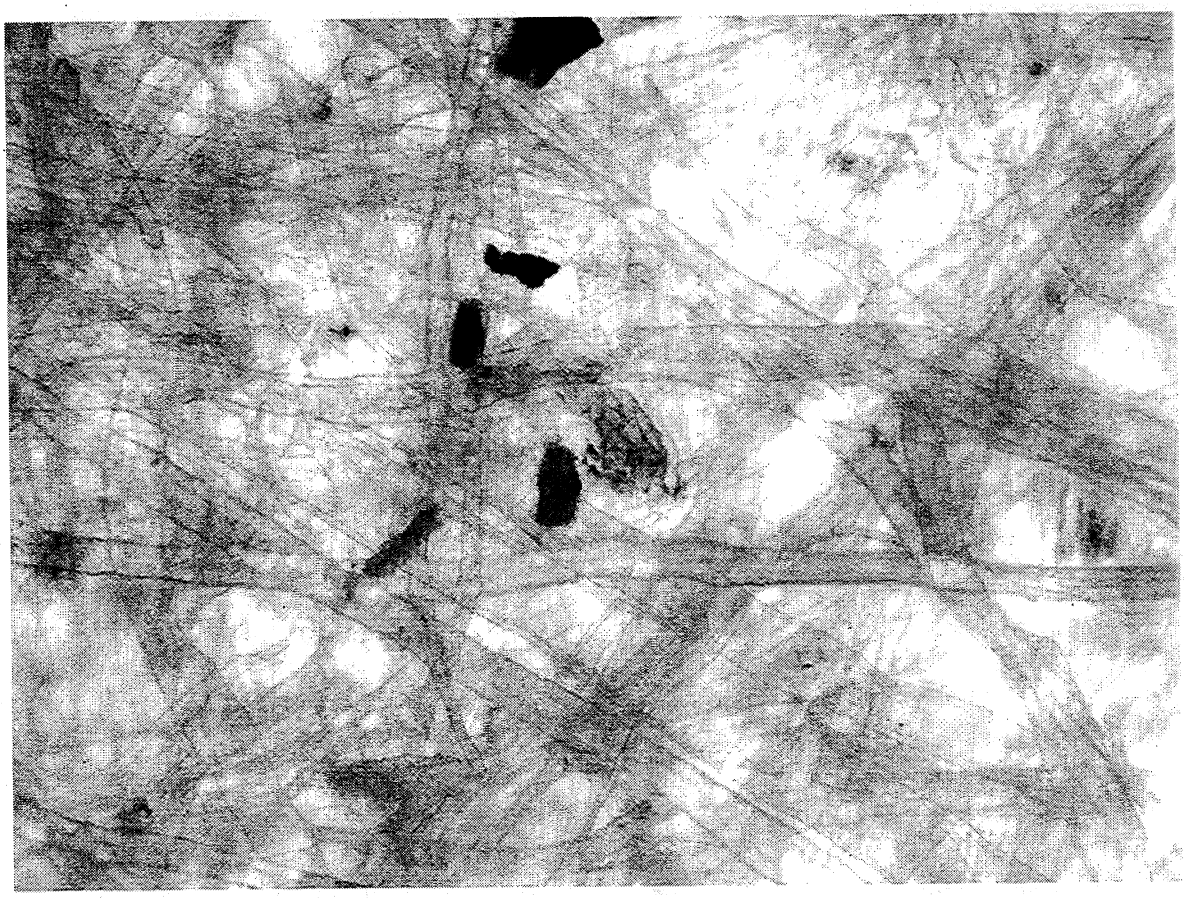

Figure 5: Sample image field under 125X magnification showing contaminants in "clean" OCC.

These findings corroborate what would be expected to remain after contaminant removal with modern screening and cleaning systems. They also confirmed that the amount of particles present in the sub-15 μm category is relatively small in number. Based upon the above findings and given the natural hydrophobicity of stickie particles, we have concluded that the most promising method of accomplishing enhanced removal of stickies from OCC streams should be through dispersed air flotation, as opposed to washing and water clarification. Further work in this laboratory will concentrate on investigating the use of flotation to remove stickies from unbleached fiber. Due to the extreme complexity and variability of "real-world" stickies, we propose to accomplish this through the use of model stickie compounds, such as those from hot melts and coating waxes [23]. We intend to report on the results of this work in a future publication.

APPENDIX

Bleaching Procedure

1. The entire bleaching procedure is performed at room temperature.
2. Weigh 133.11 grams of stock (as received).
3. Add 2000 ml of deionized water and disintegrate in British Disintegrator for 5000 rev.
4. Add an additional 500 ml of deionized water and disintegrate for another 5000 rev.
5. Add another 1000 ml of deionized water to lower the consistency.
6. Make two handsheets before bleaching to obtain a consistency reading (desired level is approximately 0.6%).
7. Collect 800 ml of slurry sample and add 67 ml of Clorox liquid bleach (approximately 12:1 ratio of 0.6% consistency stock-to-bleach).
8. Periodically stir sample while bleaching.
9. After 30 minutes, add another charge of Clorox liquid bleach (volume based on original 12:1 ratio). Stir periodically.
10. Repeat step (9) for a total of 90 minutes of bleaching (step (9) can be repeated again to make the sheet even whiter). The pH during this period was approximately 8.8.
11. Use a Britt Jar for washing. The Britt Jar should have the same mesh screen that is found in the TAPPI handsheet molds.
12. Add approximately 250 ml of bleached stock to approximately 300 ml of deionized water for washing.
13. Agitate mixture by stirring, then drain in the Britt Jar.
14. Recycle the collected liquid by pouring it back into the Britt Jar.
15. Repeat step (13).
16. Add approximately 600 ml of fresh deionized water.
17. Repeat step (13).
18. Repeat steps (16)-(17) if needed.
19. Remove collected fibers and store in a bucket.
20. Rinse edges of Britt Jar and screen with deionized water dispenser.
21. Repeat step (13) through (20) until all of the bleached mixture is washed.
22. Add deionized water to bucket to obtain approximately 0.8-1.0% consistency of bleached slurry.
23. Make handsheets in a Standard British Handsheet Mold with 80 ml of stock (a basis weight of approximately 20-25 g/m^2).
24. Place sheets on rings for drying in humidity controlled lab. (According to TAPPI standards for handsheet making).
25. Once dry; cut handsheets to fit in a petri dish. (We used a die cutter and cut our handsheets into 12.7 cm diameter handsheets that were placed in a 15 cm petri dish.)

Staining Procedure

1. Prepare a dye solution using 100 ml of 70% EtOH and 0.7 g of Sudan IV powder. The total volume can be adjusted to suit the number of handsheets to be stained. It is best to stain the handsheets with a fresh stain solution.
2. Agitate overnight (a magnet stirrer works very well).
3. Filter particulate matter using filter paper (e.g., a typical Whatman filter pad).
4. Place the handsheet in a petri dish (the handsheet should lie flat on the bottom of the petri dish).
5. Saturate the handsheet with 50% EtOH for a few seconds, and then drain the excess EtOH.
6. Apply liquid stain until section is completely covered.
7. Wrap parafilm around the petri dish.
8. Incubate sample overnight at 37°C. This should be at least 8 hr., the longer, the better.
9. It is best to perform incubation on a rotary shaker mixer or on an oscillating table to ensure the stain can easily diffuse through the sample and is not concentrated in one area of the handsheet. Alternatively, in step (6) cover the handsheet with a sufficient quantity of stain (3-5 mm in depth) to allow for easy diffusion. Periodic gentle agitation will also help.
10. After incubation, pour 50% EtOH in another petri dish and wash by dipping handsheet into the fresh 50% EtOH until no more stain leaches from the sample.

11. Repeat step (10) as often as necessary.
12. Dry the handsheet by pressing between blotter paper. When the handsheet is slightly damp, place between blotter paper and put the handsheet/blotter paper sandwich on a smooth, hard surface and press with a weight to make flat.
13. Perform image analysis by placing the handsheet on a white piece of copy paper and put a heavy circular washer around the area of interest (or a heavy glass slide) to ensure the handsheet image area is flat.
14. Use back-lighting and direct lighting to further enhance the image contrast

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